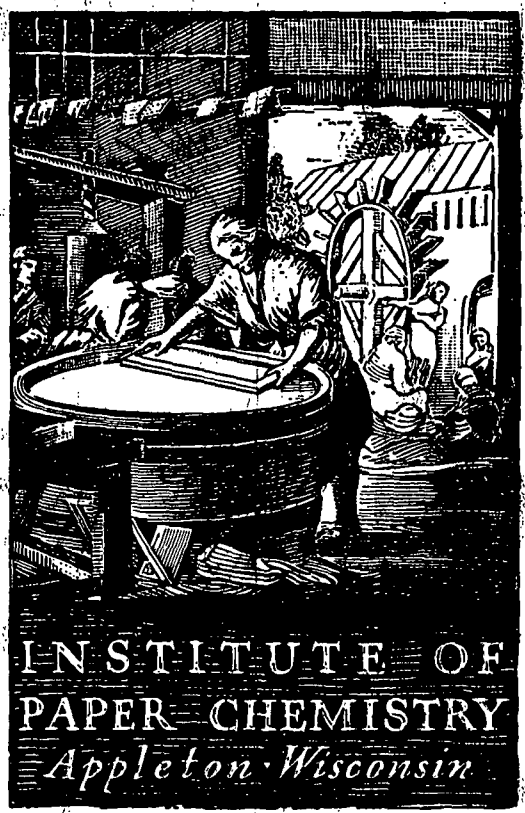

AUTOMATIC INSTRUMENTATION FOR CHEMICAL ANALYSIS OF PULP

Project 2634

Report Three

A Progress Report

to

MEMBERS OF GROUP PROJECT 2634

July 8, 1968

THE INSTITUTE OF PAPER CHEMISTRY
Appleton, Wisconsin

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THE INSTITUTE OF PAPER CHEMISTRY
Appleton, Wisconsin

AUTOMATIC INSTRUMENTATION FOR CHEMICAL ANALYSIS OF PULP

SUMMARY

The primary component of the proposed automated system for measuring residual lignin in pulp has been designed, built, and laboratory tested in simulated automatic operation. The results indicate that overall error of measurement in the final instrumental system will be about 3.5%. Design of the final instrument in prototype form is presently proceeding, along with selection of satellite components.

An automatic system for sampling digesters or blow lines for pulp is being designed. This system is based on a somewhat similar one that has been observed in semiautomatic operation for over a year, and will present to the Nu number monitor a continuous sample of screened, washed pulp at about 0.1% consistency.

INTRODUCTION

The results of a thorough investigation of various possible means of automatically measuring residual lignin in pulp have been presented in Report One, dated June 14, 1967. The essential conclusion was that the most favorable chemical approach involved reaction of the pulp with nitric acid to produce a colored compound in solution. The intensity of the color at 425 nm. was found to be a linear function of the lignin content of the pulp. Some brief experiments were reported in Report One concerning the nature of the colored substance.

Additional work has been done in an attempt to more closely identify the colored reaction product of lignin and nitric acid, but positive identification remains elusive. Since the expenditure of more time and money in this direction would contribute little to the overall objectives of the project, this work has ceased and will not be pursued further.

Report Two, dated October 12, 1967, was mainly concerned with initial experiments designed to outline the feasibility of the proposed quantitative pulp-sampling system. Using an apparatus already on hand, it was established that at room temperatures the o.d. fiber content of a compressed fiber pad is linearly related to the thickness of the pad under static load.

Based on these preliminary data, a number of smaller compressibility devices were constructed of various materials the purpose of which was to obtain data on the behavior of pads containing 100 mg. or less of fiber in preparation for the design of a final system. This report contains those data along with a description of the final design arrived at after a program of materials testing.

While difficulties were encountered in obtaining and testing suitable materials of construction, which set back the program 6-7 months, the problem is

now solved and the design of the overall system for automated measurement of lignin in pulp is proceeding. In this report are contained the details of laboratory testing in simulated automatic operation of an apparatus constructed of suitable materials in which the pulp sample can be measured and in which the Nu number test can also be carried out.

PRELIMINARY DESIGNS OF COMPRESSIBILITY APPARATUS

On page 39 of Report Two is a discussion of a pyrex-stainless steel compressibility device. This was constructed with a cylinder of 1-in. i.d. precision-bore pyrex, but could not be used because of dimensional distortions in the cylinder during the glass-blowing stage of fabrication. Professional glass-blowers advised changing the design, but it was seen that any hot working of precision-bore glass tubing would result in unacceptable distortions, so an all-stainless steel system was built.

Type 304 stainless steel resists nitric acid and can be machined to the tolerances desired for the fit of the piston in the cylinder. However, when the system was tested in the laboratory with a slurry of unbleached kraft pulp in the cylinder, the piston would not descend freely so as to form a pad of fibers. In the absence of fibers, with the cylinder containing only water, the piston moved freely the whole length of the cylinder. Changing the piston diameter to a closer or looser fit in the cylinder did not prevent the piston from jamming, nor did any improvement result from changing the piston material to Teflon.

In past studies of the compression behavior of fiber pads, an attempt was made to use an all-brass system, and the same difficulties were encountered. However, at that time it was thought to be simply a matter of closeness of fit of the piston in the cylinder so as to prevent fibers from entering space between the cylinder wall and the sides of the piston. It is known now that the fit of the piston in the cylinder is not the primary cause of piston jamming, but that some factor related to interaction of the fibers with the materials of construction is the primary cause.

Since previously successful compressibility devices have been constructed of Plexiglas (acrylic), a search was begun for a nonmetal that could be machined, that would withstand nitric acid at 85°C., and that would have the necessary physical strength and rigidity. Polysulfone, a material introduced in 1965 by Union Carbide, is a strong, rigid thermoplastic which seemed to meet the requirements. It has a thermal expansion coefficient of 3.1×10^{-5} in./in./°F. over the range -22 to +300°F., a tensile strength of 10,500 p.s.i., is nearly inert to mineral acids, alkalies, salt solutions, and most nonpolar solvents, and is easily machined.

A cylinder and piston arrangement was constructed of polysulfone, and the piston promptly jammed in the cylinder when attempts were made to form a pad of fibers in the apparatus. Again, varying the fit of the piston in the cylinder did no good, nor would metal pistons work. It was found, however, that a polysulfone piston faced with a stainless steel screen and attached to a stainless steel rod would work very nicely in a pyrex cylinder.

Since recent experience showed that precision-bore pyrex tubing could not be reheated and worked without considerable dimensional distortion, a design was chosen whereby a length of 1-in. i.d. pyrex tubing could be fitted into a polysulfone section without reheating. This assembly was so designed that it could later form part of the automated instrument.

LABORATORY TESTING OF COMPRESSIBILITY COMPONENT

DESCRIPTION

The apparatus built of pyrex and polysulfone is shown in vertical cross section in Fig. 1. The cylinder is 9 inches long, 1.0100 ± 0.0002 inches i.d., and 1.16 inches o.d. It is enclosed by a water jacket made of 2.75-inch o.d. pyrex tubing. The cylinder and water jacket are fitted together by O-ring seals into the lower polysulfone assembly and also at the top with a polysulfone cap.

The upper piston is of polysulfone faced with 80-mesh Type 304 S.S. screen fastened to the piston face with a glue made by dissolving polysulfone in methylene chloride. The piston rod is hollow down to the upper face of the piston where two diametrically opposed holes are drilled through the hollow rod wall to permit withdrawal and introduction of liquids from and to the cylinder.

The lower piston, unlike the upper one, is not porous, but is made of solid polysulfone and has a Viton O-ring to seal it against the walls of the glass cylinder when it is in the raised position. The piston rod is stainless steel, Type 304. When in the lower position, it rests in a close-fitting cavity in the lower polysulfone assembly so that its upper surface forms a tangent with the lower part of the sample and wash line. This cavity can also be flushed with water when the piston is in the raised position through the two wash nipples indicated in Fig. 1. The entry of the piston rod into the polysulfone assembly is sealed by two Viton O-rings.

The lower polysulfone assembly, into which the pyrex cylinder fits, was machined from a solid block of the plastic. The wash and sample entry nipples were also machined from polysulfone and glued into the block with polysulfone solution in methylene chloride.

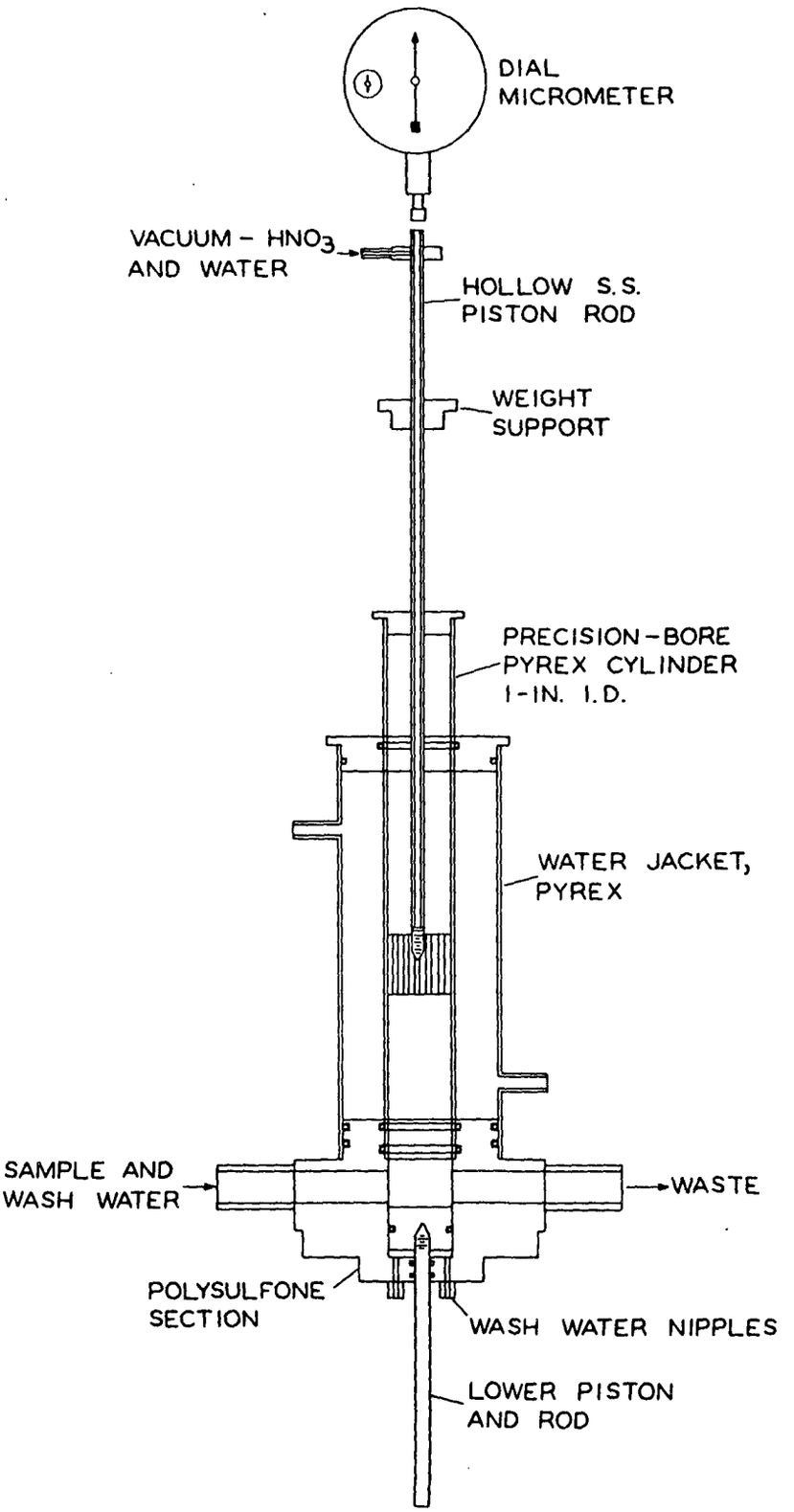


Figure 1. Cross Section of Compressibility Component

OPERATION

Although the pyrex-polysulfone compressibility assembly was designed not primarily as a manually operated laboratory device but as a component of the final automatic instrument, initial testing is necessarily by manual operation. This requires the use of satellite components not necessarily found in the final instrument. Accordingly, the apparatus was mounted on a solid brass stand and fitted with a dial micrometer for pad thickness measurements.

The sequence of operations for manual testing was as follows:

1. The bottom piston was raised to enter the pyrex cylinder at a fixed and repeatable height.

2. A measured volume of pulp slurry at about 0.1% consistency was poured into the top of the cylinder. (The cylinder capacity is about 80 cc.)

3. The upper piston (weighted) was inserted into the cylinder, lowered slowly through the slurry until a pad formed which supported the piston.

4. A timer was started and the foot of the dial micrometer placed atop the upper piston rod.

5. After three minutes' compression time, the micrometer was read and the water above the piston was drawn off through the hollow piston rod.

6. The upper piston was removed from the cylinder, 50 ml. of 16% HNO_3 were poured into the cylinder, and a timer was started.

7. The upper piston was replaced in the cylinder and slowly raised and lowered repeatedly to provide agitation.

8. After eight minutes' reaction time, the upper piston was lowered to form a pad from the fibers, and the liquid above the piston was withdrawn through the piston rod for measurement of color intensity at 425 nm.

9. With the upper piston resting on the fiber pad, the bottom piston was lowered into its cavity, and the pad was flushed to waste with water through the right nipple in the polysulfone assembly.

To calibrate the device for the pad thickness-pad weight relationship, Steps 6, 7, and 8 were omitted and, instead of flushing the fibers to waste, they were caught in a beaker, filtered off, dried, and weighed.

Sixteen percent HNO_3 was used so that, with the water remaining in the pad and in the holes in the piston, the final concentration would be 14%.

EXPERIMENTAL

Pad Weight vs. Thickness

Previous data (Report Two) relating pad weight to pad thickness were obtained with the Institute's general compressibility tester which has a cylinder i.d. of three inches. The pad weights were correspondingly large: up to 7 grams o.d. Since these amounts of pulp are several orders of magnitude in excess of the requirements of the Nu number test, the apparatus shown in Fig. 1 is scaled down to handle pads containing as little as 20 mg. o.d. pulp at thicknesses measurable with some precision.

To establish the pad weight-pad thickness relationship for this scaled-down version, an unbleached kraft pulp made from essentially 100% Douglas-fir was selected that had a Kappa number of 56.1. This is hereafter referred to as kraft Pulp no. 1.

The results listed in Table I and illustrated in Fig. 2 were obtained at room temperature (23°C.), following the sequence of operations described in the preceding section.

Using the same pulp but raising the temperature to 50°C. by circulating water from a constant-temperature bath through the water jacket, the data listed in Table II and illustrated in Fig. 3 were obtained. Similar data at 85°C. are listed in Table III and plotted in Fig. 4. In all cases the compressing pressure was 41,500 dynes/cm.^{2} (0.8 p.s.i.).

Since the regression equations for the 23°C. data and the 85°C. data are similar with regard to slope and intercept, there appears to be no significant difference in the compression response of this pulp as a result of wide temperature variations. This is illustrated in Fig. 5 where the two sets of data are plotted and their common regression line is drawn.

Two additional unbleached kraft pulps were used to establish the pad weight-pad thickness relationship at 85°C. One was made from a mixture of pine-woods that was about two thirds loblolly pine with the rest slash and longleaf pine. This pulp is referred to here as kraft Pulp no. 2, and has a Kappa number of 28.5. Kraft Pulp no. 3 was made from southern pine and has a Kappa number of 96.7.

The results for all three pulps at 85°C. are shown in Fig. 6, where their common regression line is drawn through the data points. The data for Pulps 2 and 3 are listed in Table IV. (See Table III for data for Pulp no. 1.)

TABLE I

KRAFT PULP NO. 1, PAD WEIGHT VS. PAD THICKNESS AT 23°C.

O.d. Weight, mg.	Thickness, in.
31.3	0.0178
47.3	0.0270
63.7	0.0363
62.6	0.0364
88.6	0.0549
81.6	0.0488
86.0	0.0527
128.8	0.0837
143.4	0.0923
40.8	0.0233
46.0	0.0263
70.4	0.0430
58.6	0.0358
76.5	0.0477
82.9	0.0525
87.9	0.0541
117.1	0.0750
115.9	0.0753
141.0	0.0924
105.2	0.0662

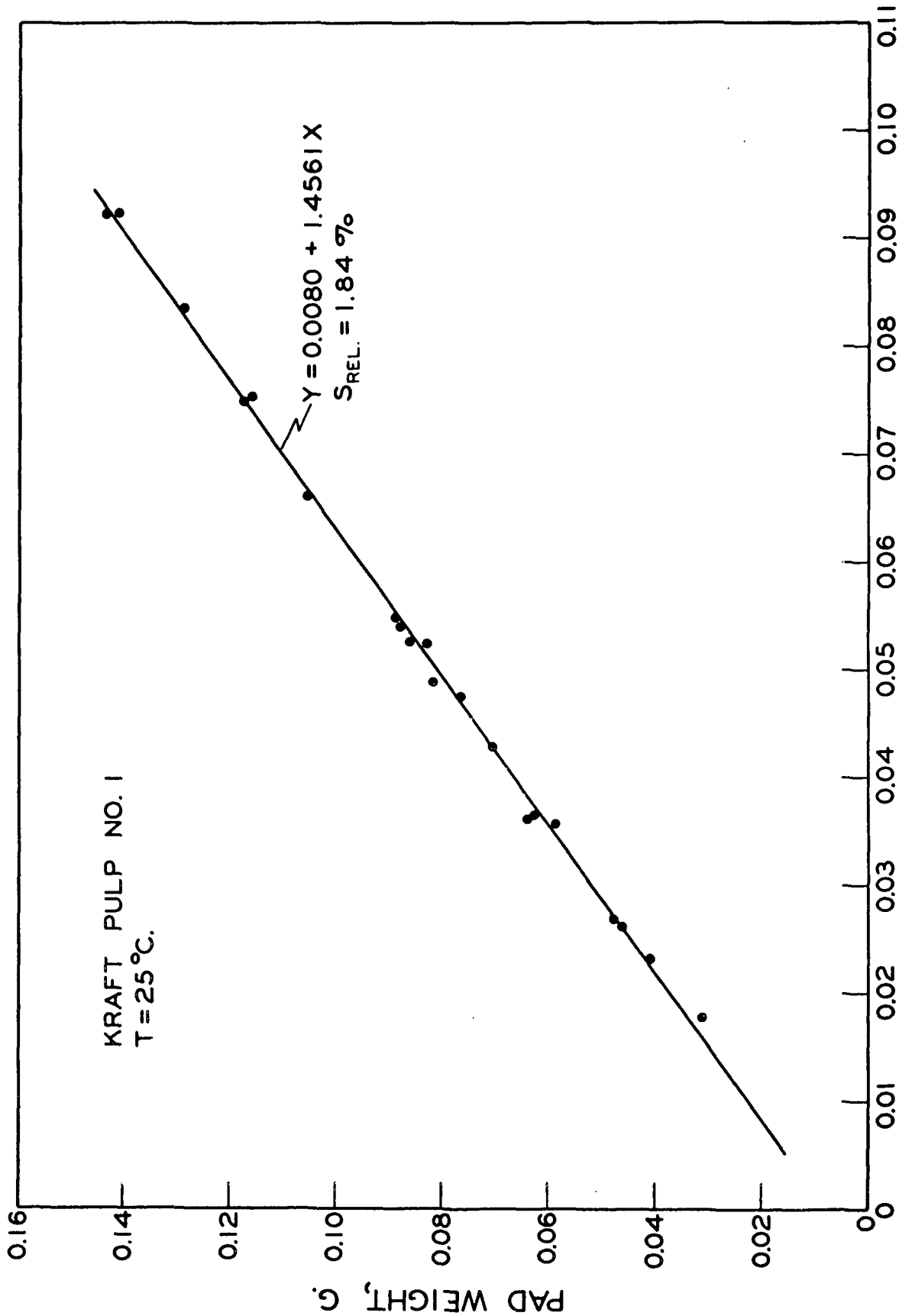


Figure 2. Pad Weight vs. Pad Thickness for Kraft Pulp No. 1 at 25°C.

TABLE II

KRAFT PULP NO. 1, PAD WEIGHT VS. PAD THICKNESS AT 50°C.

O.d. Weight, mg.	Thickness, in.
24.9	0.0147
36.0	0.0220
52.0	0.0295
81.6	0.0500
57.8	0.0342
100.0	0.0622
95.1	0.0602
108.6	0.0687
119.3	0.0767
137.7	0.0932
97.7	0.0630
34.6	0.0206
58.1	0.0357
102.0	0.0662
82.9	0.0515
64.0	0.0393
73.5	0.0735
129.6	0.0839

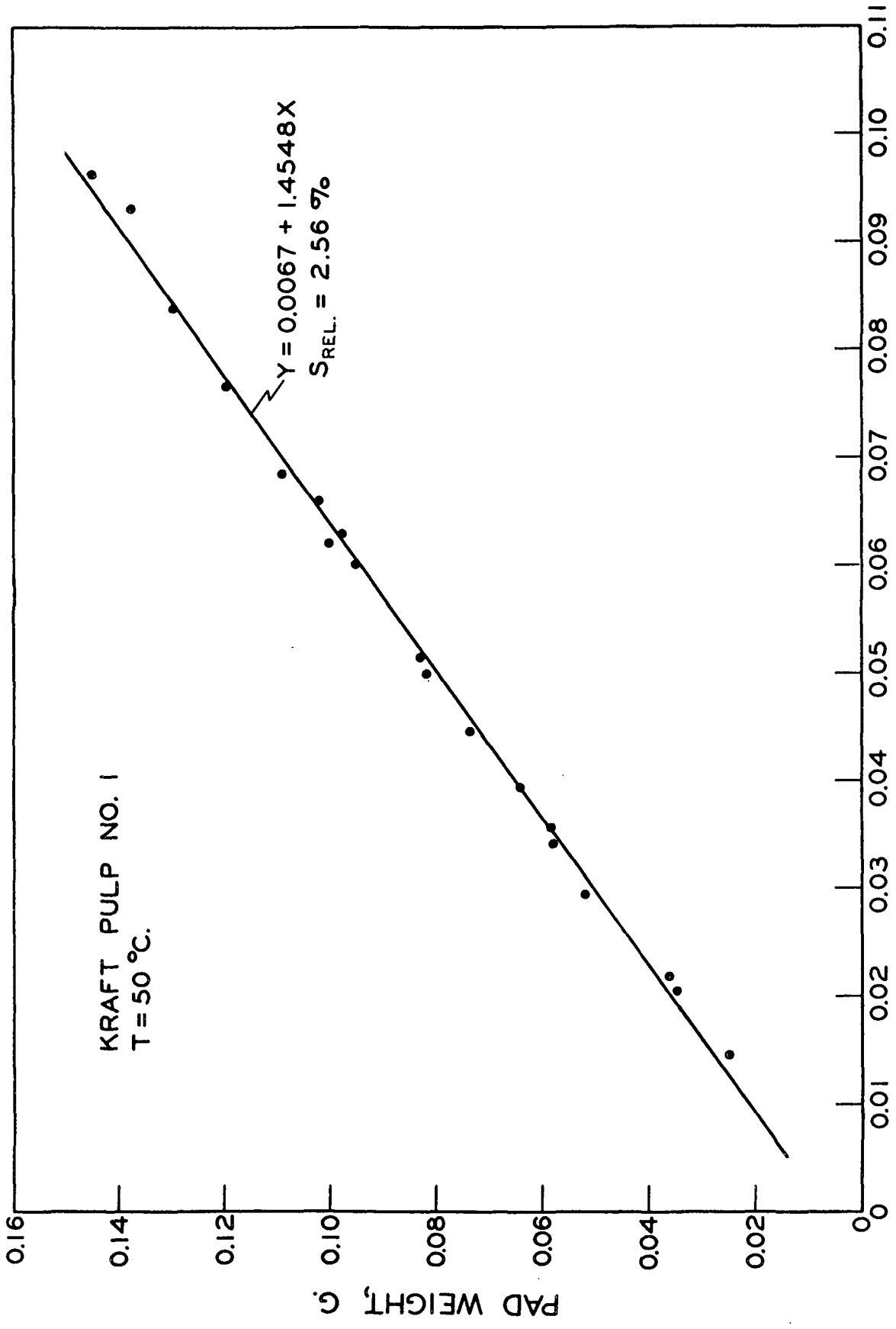


Figure 3. Pad Weight vs. Pad Thickness for Kraft Pulp No. 1 at 50°C.

TABLE III

KRAFT PULP NO. 1, PAD WEIGHT VS. PAD THICKNESS AT 85°C.

O.d. Weight, mg.	Thickness, in.
104.7	0.0631
96.2	0.0586
74.3	0.0450
62.6	0.0371
58.9	0.0348
54.6	0.0317
38.8	0.0217
39.6	0.0220
29.4	0.0162
150.3	0.0936
146.0	0.0950
136.8	0.0870
112.8	0.0700
105.8	0.0668
96.7	0.0598
103.8	0.0643
70.7	0.0443
72.5	0.0462
59.4	0.0359
32.9	0.0202

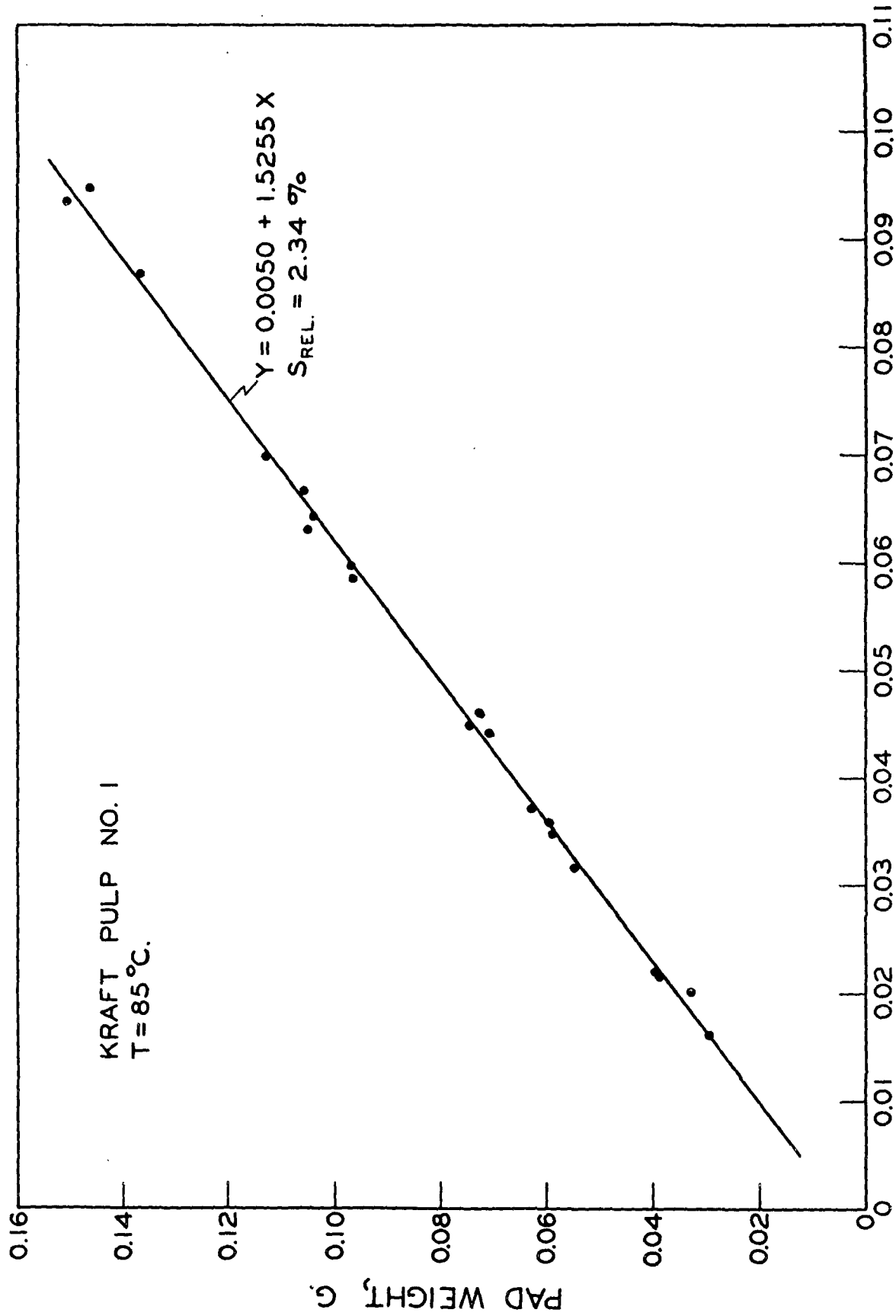


Figure 4. Pad Weight vs. Pad Thickness for Kraft Pulp No. 1 at 85°C.

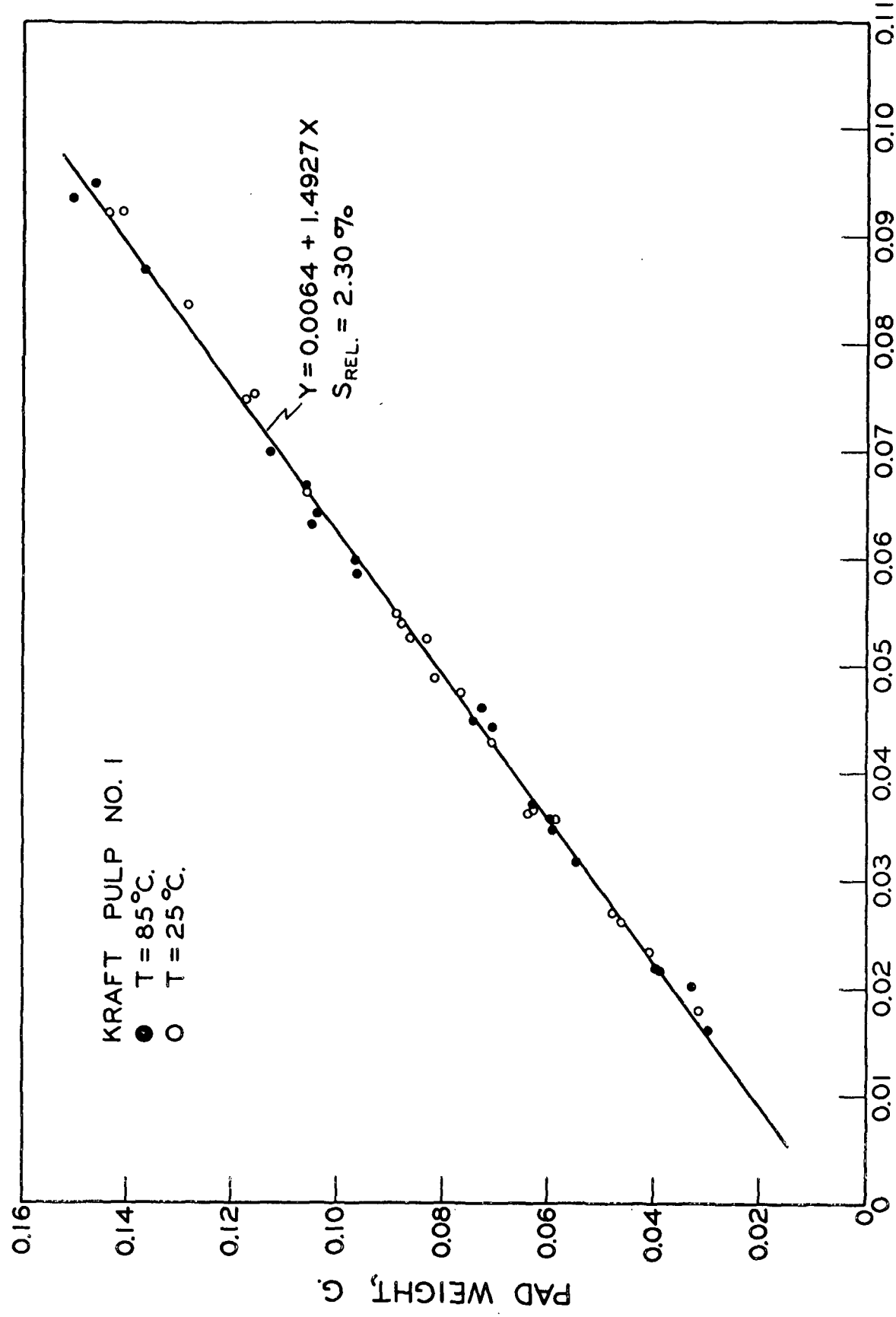


Figure 5. Pad Weight vs. Pad Thickness for Kraft Pulp No. 1 at Two Temperatures

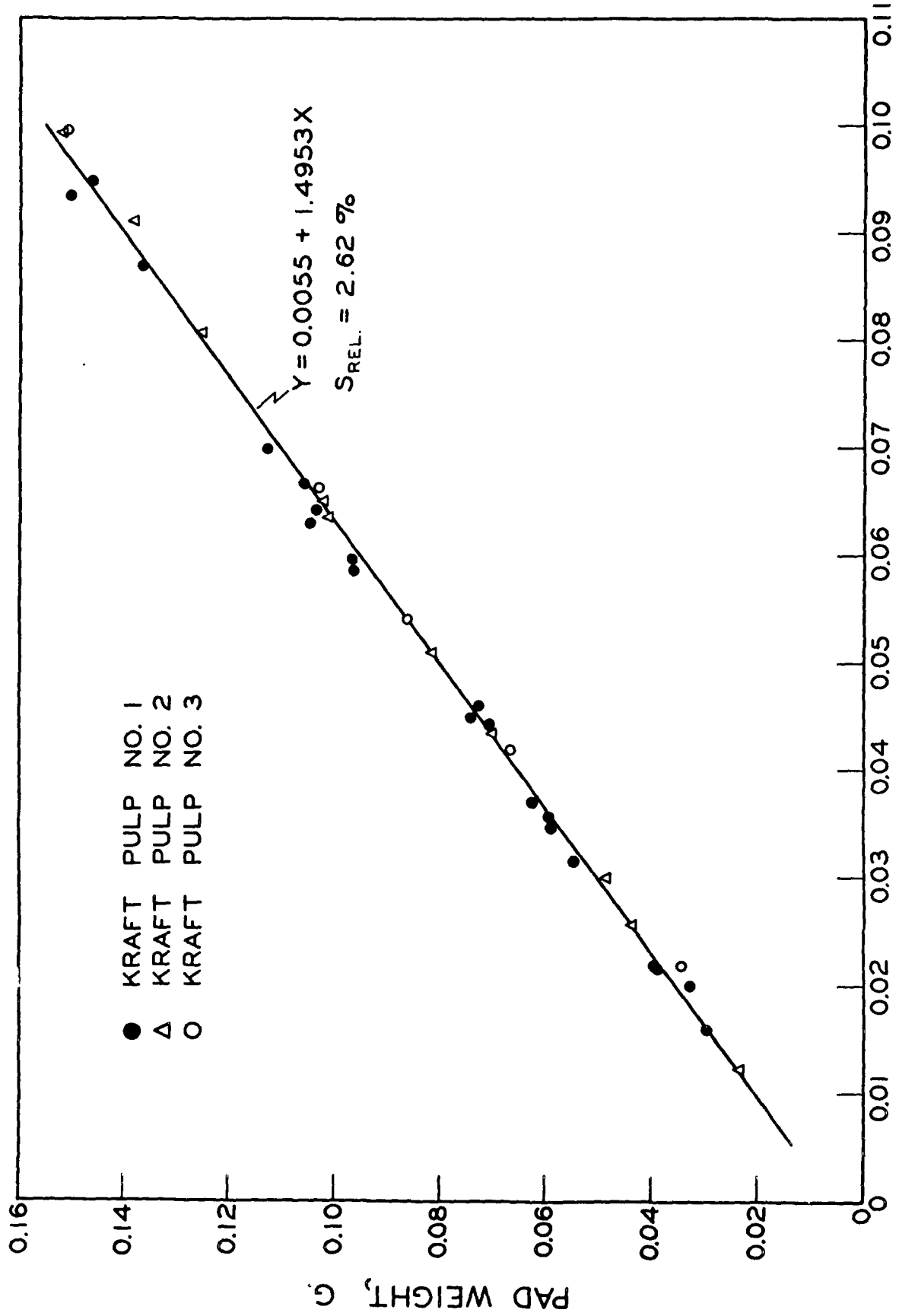


Figure 6. Pad Weight vs. Pad Thickness at 85°C. for Three Different Kraft Pulps

TABLE IV

KRAFT PULPS 2 AND 3, PAD WEIGHT VS. PAD THICKNESS AT 85°C.

	O.d. Weight, mg.	Thickness, in.
No. 2	23.4	0.0124
	43.8	0.0256
	81.9	0.0510
	101.2	0.0636
	138.3	0.0911
	48.7	0.0302
	70.3	0.0435
	102.2	0.0652
	125.2	0.0808
	151.5	0.0995
No. 3	34.1	0.0218
	66.6	0.0420
	86.1	0.0542
	103.1	0.0663
	150.6	0.0997

Measurement of Nu Number

Having established the calibration of the apparatus for pad weight vs. pad thickness at 85°C., six different samples of the three different pulp slurries were sequentially placed in the cylinder, compressed, their thickness measured, dewatered, and the Nu number of each sample determined in the system according to

the sequence previously described. At the same time, the same number of Nu number determinations were made at 85°C. on 50-mg. samples of each of the three pulps according to the manual procedure outlined on page 63, Report One.

In both series of tests the reaction time at 85°C. was 8 minutes. As shown in the reaction rate curve in Fig. 7, this is the reaction time for maximum color intensity of the yellow substance extracted from the pulp by nitric acid.

The results of all the Nu number tests are listed in Table V. The mean Nu number obtained by simulated automatic operation is plotted against Kappa number in Fig. 8.

CONCLUSIONS AND DISCUSSION

The data presented in Tables I-IV and the relationships illustrated in Fig. 2-6 solidly demonstrate that o.d. pad weights can be estimated from a measurement of the compressed pad thickness with an error of about 2.5%. It is further shown that temperature variations of as much as 60°C. have little effect on the accuracy within this limit. Three very different kraft pulps respond similarly in the system, and, in fact, have the same regression line for pad weight vs. pad thickness.

The Nu numbers listed in Table V under "Automatic Procedure" were calculated according to Equation (1):

$$Nu = \frac{100 \times A_s \times 30}{A_{std.} \, W_s} \qquad (1)$$

where

A_s = absorbance at 425 nm. of yellow HNO_3 extract of pulp,

$A_{std.}$ = absorbance at 425 nm. of standard aqueous solution of 100 mg./liter $K_2Cr_2O_7$, and

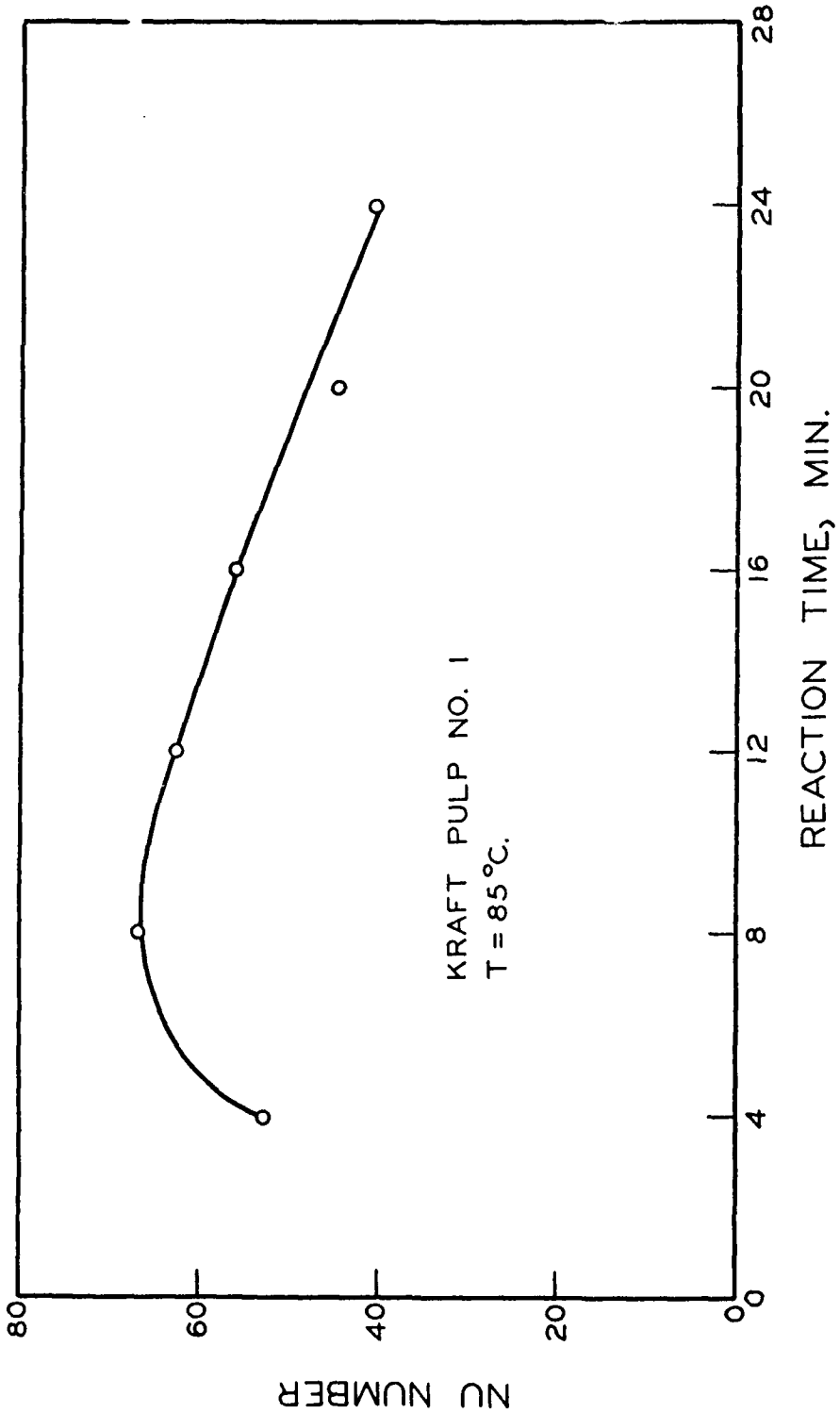


Figure 7. Reaction Rate Curve of Kraft Pulp No. 1 with 14% HNO_3

TABLE V

NU NUMBER BY SIMULATED AUTOMATIC PROCEDURE AND STANDARD MANUAL PROCEDURE

	Automatic Procedure		Manual Procedure	
	Sample, mg.	Nu Number	Sample, mg.	Nu Number
		Kraft Pulp No. 1		
	51.4	72.0	50.1	66.2
	49.9	76.7	50.9	66.2
	49.2	74.4	48.0	67.1
	49.2	74.8	49.8	62.1
	48.6	74.2	48.9	64.0
	44.1	76.4	48.5	65.7
Mean:	48.7	74.8	49.4	65.2
$s_{rel.}$:	4.6%	2.1%	2.0%	2.6%
		Kraft Pulp No. 2		
	53.8	37.6	50.2	29.9
	53.3	37.6	49.4	28.9
	51.4	36.9	49.4	33.0
	51.1	38.2	49.3	28.2
	51.0	37.5	49.3	29.3
	49.5	37.9		
Mean:	51.7	37.6	49.5	29.9
$s_{rel.}$:	2.8%	1.1%	0.7%	5.6%
		Kraft Pulp No. 3		
	53.3	120	56.5	117
	52.8	121	51.7	117
	52.3	122	51.6	117
	50.8	125	51.4	121
	49.9	123	50.2	119
	49.6	121	49.5	116
Mean:	51.5	122	51.8	118
$s_{rel.}$:	2.8%	1.3%	4.3%	1.4%

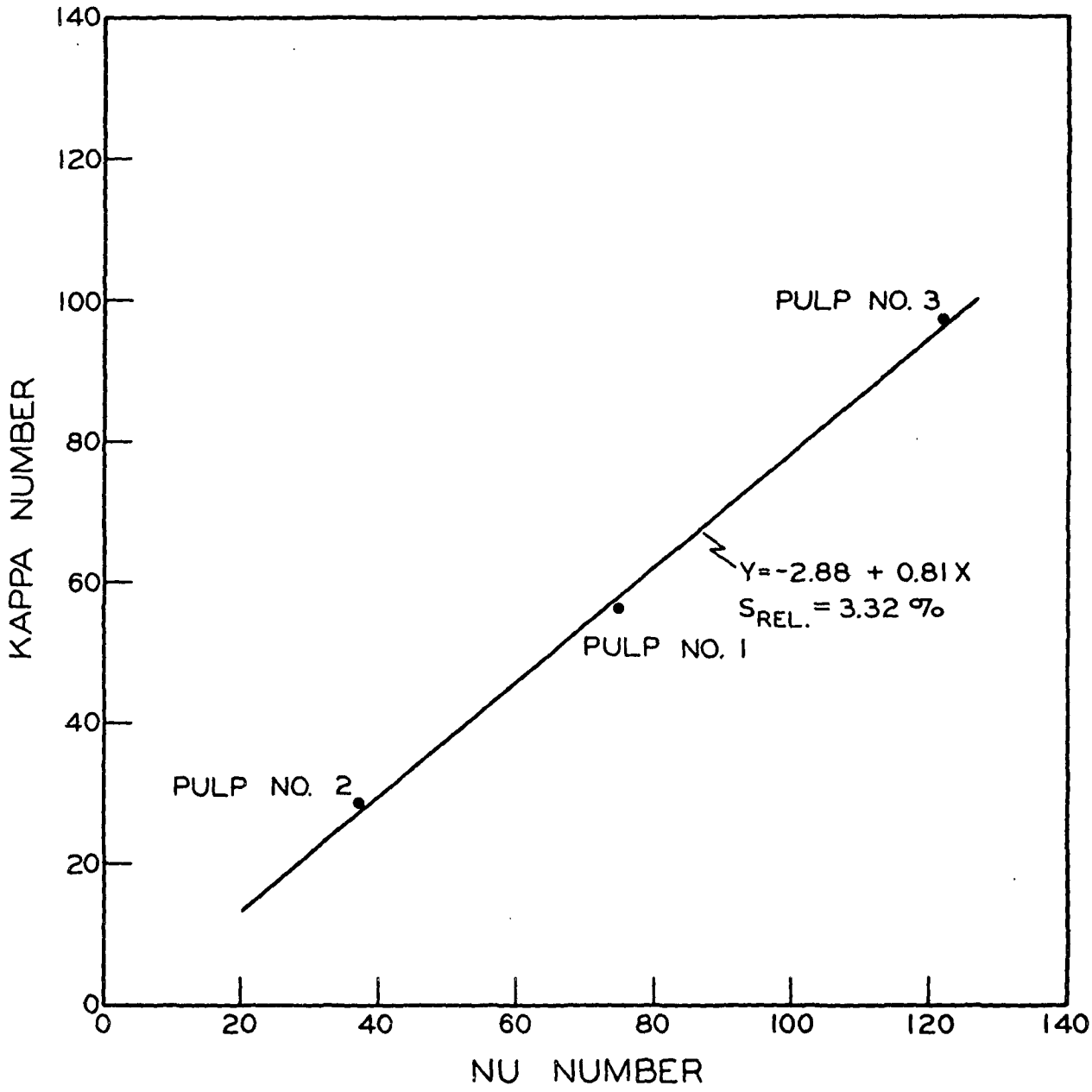


Figure 8. Kappa Number vs. Nu Number for Three Different Kraft Pulps on Automatic Compressibility Component

$\underline{W_s}$ = weight of pulp sample in milligrams.

In this case, $\underline{W_s}$ was calculated from the regression equation shown in Fig. 6. Consequently, the relative error of 3.32% in the Kappa-Nu relationship shown in Fig. 8 is the overall error of the whole procedure involving measurement of both $\underline{W_s}$ and $\underline{A_s}$. Even though only three different pulps are represented, the three data points in Fig. 8 represent 18 experiments, a population considered sufficient to establish with some validity the overall Kappa-Nu relationship on the apparatus shown in Fig. 1.

It can be seen in Table V that the Nu numbers measured by the "Automatic Procedure" are consistently higher than those derived from the "Manual Procedure." This difference is most likely due to easily observed variations between the two procedures. In the manual procedure, the dried pulp is weighed into a beaker, the nitric acid is added, and the beaker is placed in a water bath. The sample, as it is weighed out, is torn into bits from a pad of the pulp, and while the beaker sits in the water bath there is no stirring except for an occasional swirling of the beaker.

On the other hand, in the automatic apparatus the pulp is well disintegrated down to individual fibers, and there is constant agitation by the upper piston.

This interpretation is reinforced by the results shown in Table V for kraft Pulp no. 2. This pulp was available as a dried pad which was very difficult to disintegrate and was very resistant to water penetration. The relative error of the manual Nu numbers, 5.6%, is the highest of the three, while, after it was finally disintegrated for the "Automatic Procedure," the relative error of 1.1% was the lowest of the three.

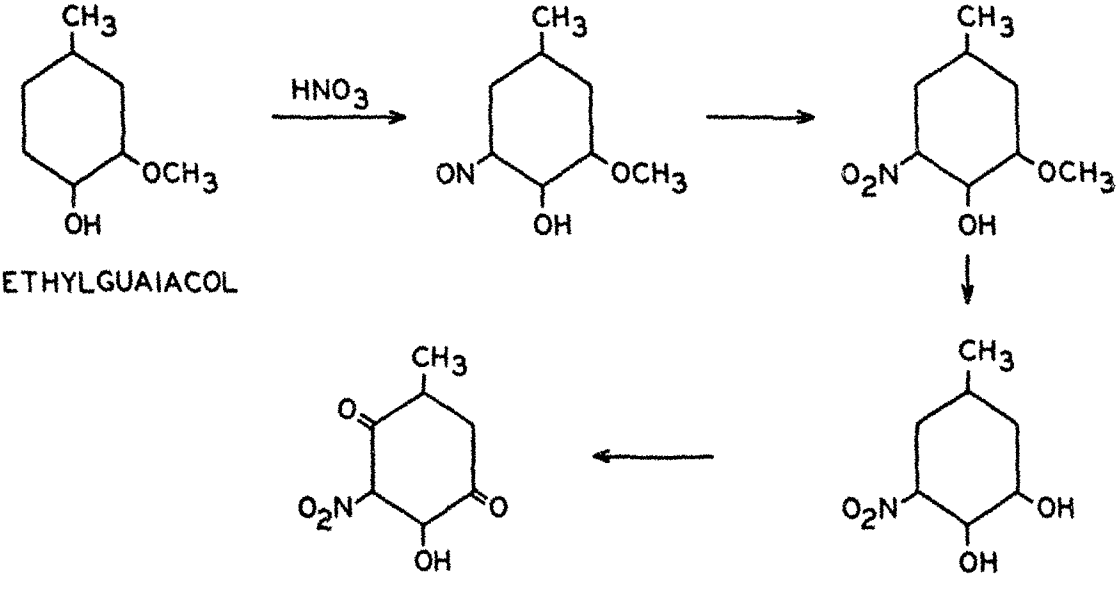
In general, these results show that the system for sample measurement and Nu number determination, as shown in Fig. 1, will yield results of acceptable precision and reliability for process control. At this stage of development, it appears that once the sample of pulp slurry at about 0.1% consistency is placed in the cylinder, the residual lignin in that pulp expressed as Kappa or Nu number will be known 12-18 minutes later. This means that the overall instrument will yield three to five such measurements per hour.

NATURE OF THE YELLOW SUBSTANCE EXTRACTED FROM PULP WITH NITRIC ACID

The basis for the Nu number test is the measurement at 425 nm. of the absorbance of the solution resulting from the reaction of pulp and nitric acid. An attempt was made to more closely identify the species which absorbs at 425 nm.

Thirty-mg. samples of pulp were weighed into 100-ml. beakers. The samples were treated with 50 ml. of 5, 10, 14, 20, 30, 40, 50, 60, and 70% nitric acid for 30 minutes at 70°C. After reaction, each solution was scanned from 600 to 320 nm. When nitric acid of 40% concentration or lower was used, the scans had shoulders at 425 nm. When the nitric acid concentration was greater than 40%, the scans had no shoulders at 425 nm. In this case, the absorbance at 425 nm. was due to the broad peak centered around 345 nm. As the nitric acid concentration increased, the shoulder at 425 nm. decreased (Fig. 9) and the peak at 345 became broader. Kalisch (1) states that high nitric acid concentrations favor oxidation and low concentrations favor nitration. Therefore, the species absorbing at 425 nm. must be a nitration rather than an oxidation product.

Sobolev (2) nitrated 4-methylguaiacol in organic solvents and identified the intermediate and final products:



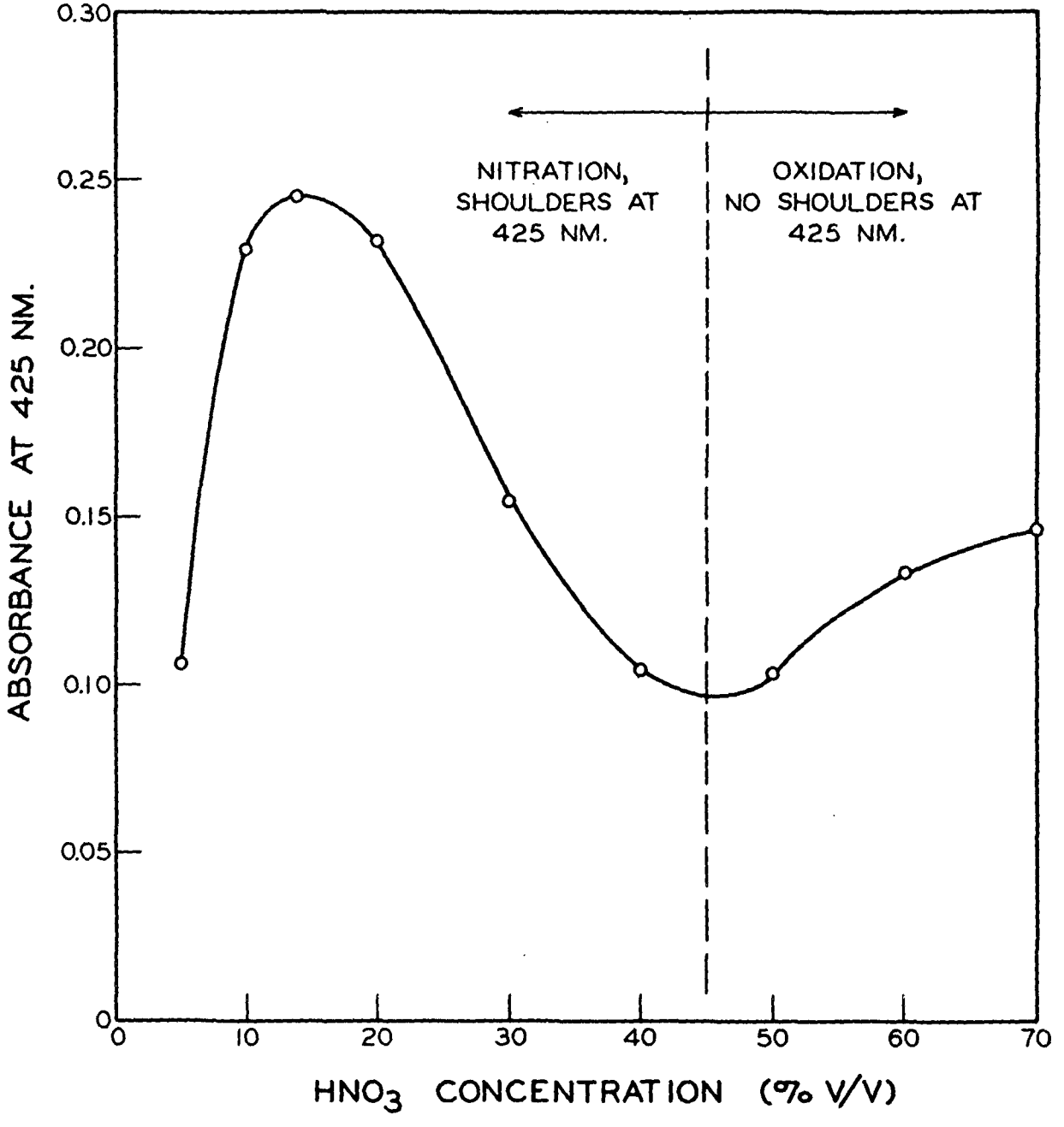
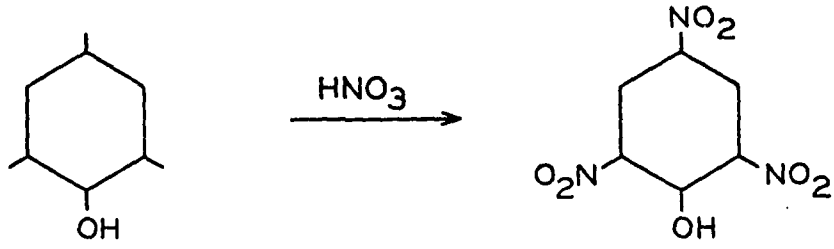


Figure 9. Absorbance at 425 nm. of Product From Reaction of Nitric Acid With Unbleached Kraft Pulp, as a Function of Nitric Acid Concentration

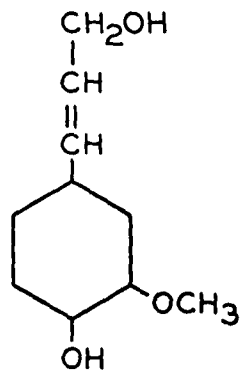
It was thought that nitration without an organic solvent might result in a substitution of a nitrate group for the side chain. Picric acid is often a product of nitration of phenolic compounds:



PICRIC ACID

A solution of picric acid was scanned from 600 to 320 nm. The scan showed no shoulder at 425 nm. and had a broad peak centered around 350 nm. Picric acid is probably formed in the reaction of pulp and nitric acid, but it seems as if the species absorbing at 425 nm. is a nitration product similar to one of the intermediates isolated by Sobolev with the side chain or chains intact.

The species absorbing at 425 nm. also appears to be similar to the nitration product of the reaction of coniferyl alcohol with HNO_3.



Coniferyl alcohol

One mg. of coniferyl alcohol was reacted with 50 ml. of 14% HNO_3 for 15 minutes at 70°C. A scan of the resulting yellow solution showed a shoulder at 425 nm. Figure 10 shows scans of pulp-HNO_3 and coniferyl alcohol-HNO_3 solutions. The scans are nearly identical.

In summary, it appears that the species absorbing at 425 nm. is a nitration product from the reaction of a lignin unit with nitric acid, similar to coniferyl alcohol. It most likely is a nitro derivative of the type isolated by Sobolev with the side chains intact.

Positive identification requires the isolation of the species. Efforts to isolate the compound have been unsuccessful. Nitric acid interferes with paper chromatography, UV, and IR. Solvent extraction fails to remove the species from the nitric acid solution. Ion exchange will neutralize and remove NO_3^- from the solution; however, the color of the solution is also destroyed.

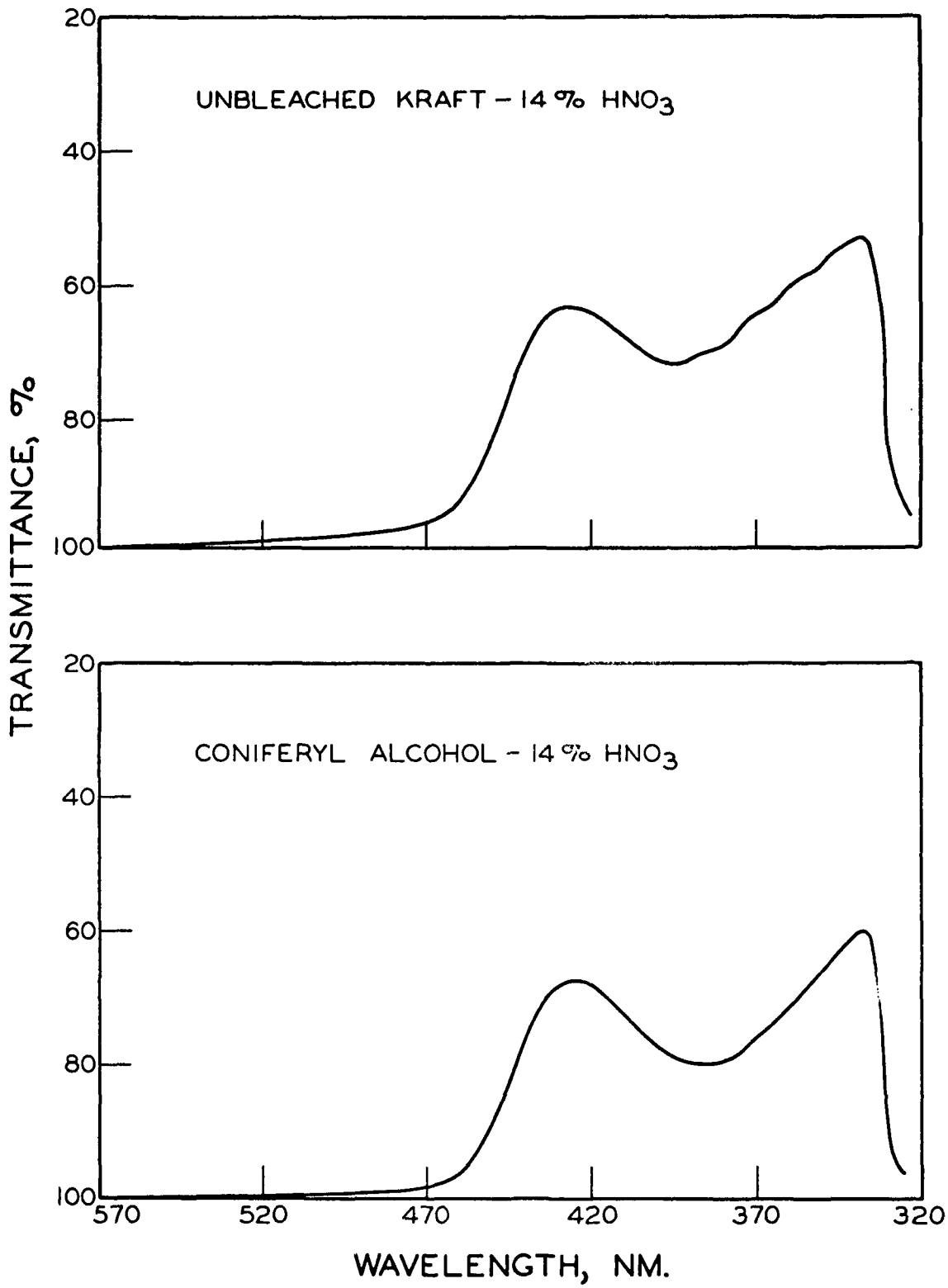


Figure 10. Absorbance Spectra of Products From Reaction of Nitric Acid With an Unbleached Kraft Pulp and With Coniferyl Alcohol

FUTURE WORK

PULP SAMPLING SYSTEM

The pulp slurry entering the cylinder of the compressibility component of the Nu number monitor must be:

1. about 0.1% consistency,
2. screened, and
3. washed.

To meet these conditions, it was originally intended only to sample from the flat-screened stock with subsequent consistency regulation prior to placing a fixed volume of slurry in the compressibility cylinder. This intention arose not from theoretical deliberations about the best place in a pulp manufacturing system to place such an instrument, but simply from the lack of a suitable system for sampling from a digester or blow line and means for preparing the sample in a reasonable time to meet the above three conditions.

However, it now appears possible to devise an automatic system for sampling pulp from a batch digester near the end of the cook, from a blow line, or from the bottom of the lower cooking zone of a continuous digester. The system would further prepare the sample for acceptance by the Nu number monitor through a sequence of washing, screening, and consistency regulation. Such a system is presented schematically in Fig. 11.

A system similar to that shown in Fig. 11, but which is semiautomatic and stops at the point where the pulp is collected on a 65-mesh wire, has been observed in operation for over a year. To complete the system, provisions must be added for washing the pulp on the 65-mesh screen, thickening the pulp, and reslurrying

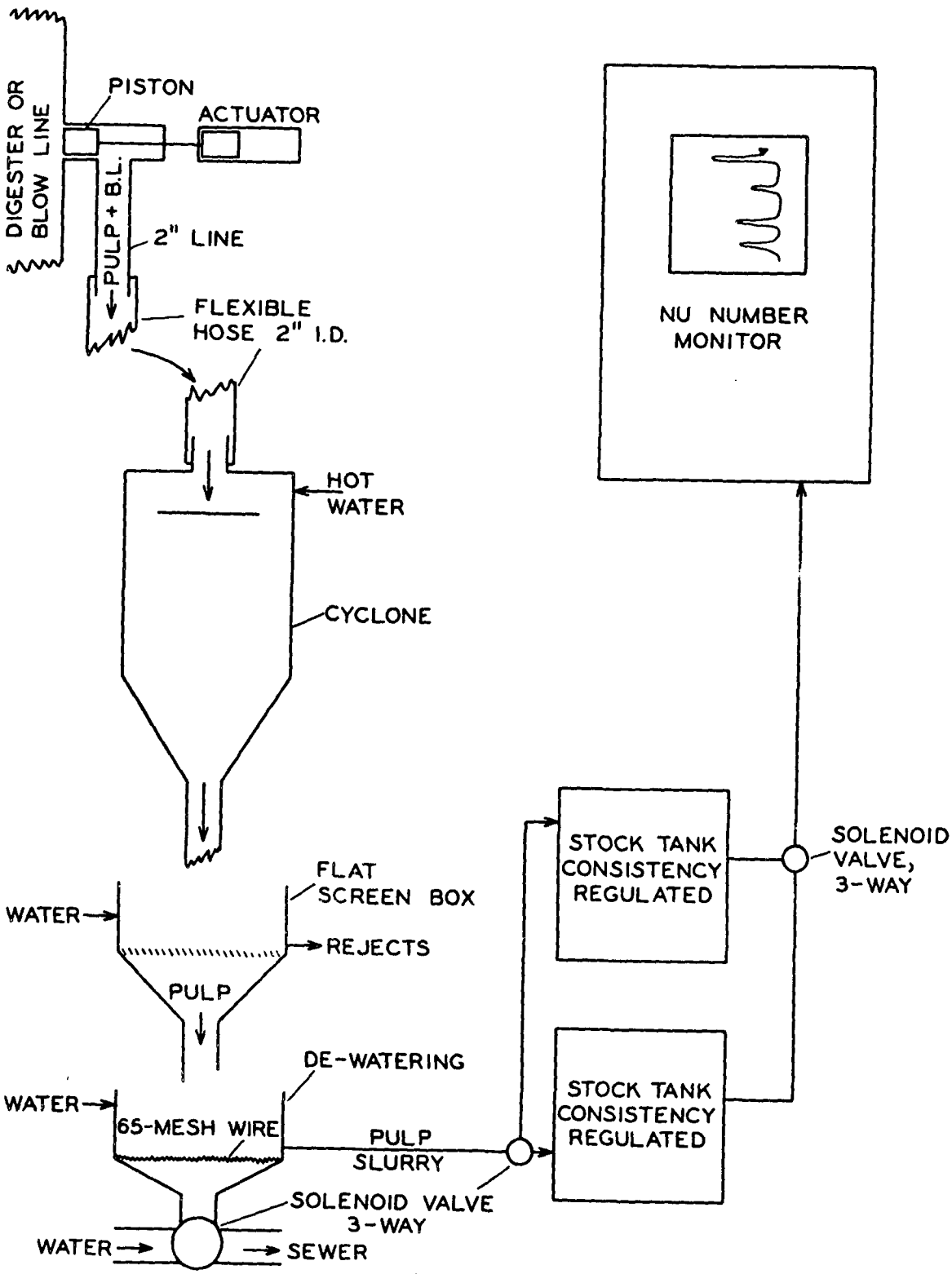


Figure 11. Proposed Pulp Sampling System

it to an approximately known consistency to feed to the stock tanks for accurate (± 5%) consistency regulation. Dual stock tanks are shown in Fig. 11, and these are intended to be used alternately so as to provide a constant supply of slurry to the Nu number monitor while fresh sample is in preparation.

NU NUMBER MONITOR

Since the compressibility component of the overall instrument has been shown to operate satisfactorily in the laboratory in simulated automatic operation, it now remains to select the satellite components for complete automatic operation, fix the sequence of events during operation, design the programmer, build the instrument, and test it.

It is intended that building of the instrument shall proceed concurrently with design and component selection for the type of pulp sampling shown in Fig. 11. However, the instrument itself will be of prime concern because it must be shown to operate satisfactorily in the laboratory before any mill installations can be made. In the laboratory, of course, it can be quite easily supplied with pulp slurry of the required properties.

It can be seen that the instrument must be provided with internal means of computation since the final output (Nu number or Kappa number) is calculated from two internal measurements: (1) pad thickness and (2) light absorbance at 425 nm. This will take the form of a simple analog "computer" capable of accepting two d.c. voltage signals, computing from these the Nu number, and putting out the result in the form of a d.c. voltage. The output will, of course, be available for acceptance by a digital print-out device, a recording potentiometer, or a process-control computer. It is intended to design the instrument so that the internal analog "computer" is optional for those mills having process-control computers.

ACKNOWLEDGMENT

The contributions of Arne Henriksen, former Research Fellow at the Institute but now with the Norwegian Institute for Water Research in Oslo, are acknowledged with thanks. Mr. Henriksen is largely responsible for development of the Nu number test, and contributed greatly to development of the compressibility component.

The assistance of Mr. Donald Sachs and Mr. John Taggart in performing much of the laboratory work is also recognized with appreciation for their hard work.

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